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IS 7067 (1973): Fluoboric acid and metal fluoborates for electroplating [CHD 5: Electroplating Chemicals and Photographic Materials]



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Indian Standard
SPECIFICATION FOR
FLUOBORIC ACID AND METAL
FLUOBORATES FOR ELECTROPLATING

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BUREAU OF INDIAN STANDARDS

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Indian Standard

SPECIFICATION FOR FLUOBORIC ACID AND METAL FLUOBORATES FOR ELECTROPLATING

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Indian Standard
SPECIFICATION FOR
FLUOBORIC ACID AND METAL
FLUOBORATES FOR ELECTROPLATING

0. FOREWORD

0.1 This Indian Standard was adopted by the Indian Standards Institution on 28 September 1973, after the draft finalized by the Electroplating Chemicals Sectional Committee had been approved by the Chemical Division Council.

0.2 Fluoboric acid is used for cleaning and etching of metal surfaces due to the high solubility of oxide and silicate films that may be on the metal surfaces. It is also used for cleaning lead and its alloys prior to electroplating. The metal fluoborates show their best advantage in plating applications in which throwing power is not critical but speed of deposition and production of high quality deposits are essential. Such applications include plating of wire and strip and electrical components used in highly corrosive conditions, electroforming, electrotyping and stereotyping, production of bearings, manufacture of printed circuits and tin or tin-lead plating for soldering. Along with deposition speed, other advantages of fluoborate plating include: approximately 100 percent anode and cathode efficiency; fine grain deposits; ease of control; high conductivity, hence lower power costs; good covering power; ease of make up of solution; simplicity and stability of solutions. The disadvantages include lack of throwing power; tendency for treeing; and high initial cost per litre of solution.

0.3 In the formulation of this standard, due weightage has been given to international coordination among the standards and practices prevailing in different countries in addition to relating it to the practices in the field in this country. This has been met by deriving assistance from draft BS:2657 'Fluoroboric acid and metallic fluoroborates for electroplating', issued by the British Standards Institution.

0.4 This standard contains clauses **3.1** and **B-2.4** which provide for agreement between the purchaser and the supplier.

0.5 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS:2-1960*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

1. SCOPE

1.1 This standard prescribes the requirements and methods of sampling and test for fluoboric acid and the following metal fluoborates for electroplating:

- a) Lead fluoborate,
- b) Tin fluoborate,
- c) Copper fluoborate,
- d) Zinc fluoborate, and
- e) Sodium fluoborate.

2. REQUIREMENTS

2.1 Description — Fluoboric acid and metal fluoborates shall conform to the following description:

| <i>Name of Material</i> | <i>Description</i> |
|-------------------------|---|
| Fluoboric acid | Clear, colourless or nearly colourless aqueous solution of fluoboric acid (HBF_4) |
| Lead fluoborate | Clear, colourless or nearly colourless aqueous solution of lead fluoborate $\text{Pb}(\text{BF}_4)_2$ |
| Tin fluoborate | Clear, colourless or nearly colourless aqueous solution of stannous fluoborate $\text{Sn}(\text{BF}_4)_2$ |
| Copper fluoborate | Clear, blue aqueous solution of cupric fluoborate $\text{Cu}(\text{BF}_4)_2$ |
| Zinc fluoborate | Clear, colourless or nearly colourless aqueous solution of zinc fluoborate $\text{Zn}(\text{BF}_4)_2$ |
| Sodium fluoborate | Colourless crystalline powder, free from dirt, foreign matter and visible impurities and corresponding essentially to the formula NaBF_4 |

*Rules for rounding off numerical values (*revised*).

2.2 Fluoboric acid and metal fluoborates shall also comply with the requirements given in Table 1 when tested in accordance with the methods prescribed in Appendix A. Reference to the relevant clauses of Appendix A is given in col 9 of the table.

3. PACKING AND MARKING

3.1 Packing—The material shall be packed as agreed to between the purchaser and the supplier.

3.2 Marking—The packages shall be marked with the following:

- a) Name of the material and its net mass;
- b) Name of the manufacturer and his recognized trade-mark, if any; and
- c) Lot number and date of manufacture.

3.3 BIS Certification Marking

The product may also be marked with Standard Mark.

3.3.1 The use of the Standard Mark is governed by the provisions of the Bureau of Indian Standards Act, 1986 and the Rules and Regulations made thereunder. The details of conditions under which the licence for the use of Standard Mark may be granted to manufacturers or producers may be obtained from the Bureau of Indian Standards.

4. SAMPLING

4.1 A representative sample of the material (solutions) of not less than 250 ml is necessary for the purpose of examination under this standard. The sample shall be packed in a suitable closed, clean dry plastics bottle fitted with a rubber or plastics stopper.

4.2 Representative sample of sodium fluoborate shall be drawn and adjudged as prescribed in Appendix B.

A P P E N D I X A

(Clause 2.2)

ANALYSIS OF FLUOBORIC ACID AND METAL FLUOBORATES FOR ELECTROPLATING

A-1. QUALITY OF REAGENTS

A-1.1 Unless specified otherwise, pure chemicals and distilled water (*see* IS: 1070-1960*) shall be used in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

*Specification for water, distilled quality (*revised*).

TABLE 1 REQUIREMENTS FOR FLUOBORIC ACID AND METAL FLUOBORATES

(Clauses 2.2 and B-4.2)

| SL No. | CHARACTERISTIC | FLUOBORIC ACID | LEAD FLUOBO- RATE | TIN FLUOBO- RATE | COPPER FLUOBO- RATE | ZINC FLUOBO- RATE | SODIUM FLUOBO- RATE | METHOD OF TEST |
|-----------|---|-------------------|-------------------------|------------------------|---------------------------|-------------------------|---------------------------|-------------------|
| (1) | (2) | (3) | (4) | (5) | (6) | (7) | (8) | (9) |
| i) | Fluoboric acid (as HBF_4), g/l | 520 <i>Min</i> | 35 <i>Max</i> | — | 35 <i>Max</i> | 20 <i>Max</i> | — | A-2 |
| ii) | Free boric acid (H_3BO_3), g/l | 10 to 60 | 10 to 60 | 10 to 60 | 10 to 60 | 10 to 60 | — | A-3 |
| iii) | Lead (as Pb), g/l | — | 500 <i>Min</i> | — | — | 0.5 <i>Max</i> | — | A-4 |
| iv) | Tin (as Sn), g/l, <i>Min</i> | — | — | 300 | — | — | — | A-5 |
| v) | Copper (as Cu), g/l | — | — | 0.05 <i>Max</i> | 200 <i>Min</i> | 0.5 <i>Max</i> | — | A-6 |
| vi) | Sulphate (as SO_4), g/l, <i>Max</i> | 5.0 | — | 5.0 | 5.0 | 5.0 | 0.05* | A-7 |
| vii) | Silica (as SiO_2), g/l, <i>Max</i> | 10.0 | 10.0 | 10.0 | 10.0 | 10.0 | 0.8* | A-8 |
| viii) | Iron (as Fe), g/l, <i>Max</i> | 0.5 | 1.0 | 2.0 | 2.0 | 1.0 | 0.5* | A-9 |
| ix) | Heavy metals other than iron, g/l, <i>Max</i> | 1.0 | — | — | — | — | — | A-10 |
| x) | Chloride (as Cl), g/l, <i>Max</i> | 0.1 | — | 1.0 | — | — | 0.03* | A-11 |
| xi) | Zinc (as Zn), g/l, <i>Min</i> | — | — | — | — | 200 | — | A-12 |
| xii) | Sodium fluoborate (as NaBF_4), <i>Min</i> | — | — | — | — | — | 96.6* | A-13 |
| xiii) | Volatile matter, <i>Max</i> | — | — | — | — | — | 0.5* | A-14 |

NOTE — Free fluoboric acid in tin fluoborate concentrates is not amenable to simple chemical analysis owing to hydrolysis. As, however, concentrates with widely different free fluoboric acid contents all appear equally satisfactory, when used in electroplating baths, no limits for free acid are prescribed in this specification.

*These values are on percent by mass basis as sodium fluoborate is in the form of crystalline powder.

A-2. DETERMINATION OF FLUOBORIC ACID

A-2.0 General—Two methods are prescribed. The first is meant for fluoboric acid content in fluoboric acid and the second is for fluoboric acid content in metal fluoborates.

A-2.1 Reagents

A-2.1.1 Calcium Chloride Solution—100 g/l. Dissolve 10 g of calcium chloride ($\text{CaCl}_2 \cdot 6\text{H}_2\text{O}$) in 100 ml of water.

A-2.1.2 Methyl Orange Indicator Solution—prepared by dissolving 0.1 g of methyl orange in 100 ml of water.

A-2.1.3 Standard Sodium Hydroxide Solution—approximately 1 N.

A-2.1.4 Standard Sodium Hydroxide Solution—approximately 0.5 N.

A-2.2 Procedure

A-2.2.1 For Fluoboric Acid—Transfer 10.0 ml of the sample to a 100-ml volumetric flask and dilute to the mark with water. Transfer a 10.0-ml aliquot to a 500-ml conical flask, add 50 ml of calcium chloride solution, 50 ml of water and a few drops of methyl orange indicator solution and titrate with standard sodium hydroxide solution (see A-2.1.3) to the first sign of colour change from pink to yellow. Cover and heat to boiling, stand on a water bath for 10 minutes and titrate to the end point. Repeat this step until the increased titration is less than 2 ml. Finally cover and boil gently for 30 minutes, cool and titrate to neutralization. Reserve this solution for determination of free boric acid and record all titrations. Let the total volume of standard sodium hydroxide solution consumed be A ml.

A-2.2.1.1 Calculation

$$\text{Free fluoboric acid (HBF}_4\text{), g/l} = A \times 21.96 \times N$$

where

N = normality of standard sodium hydroxide solution.

A-2.2.2 For Metal Fluoborates

A-2.2.2.1 Copper fluoborate—Transfer 10.0 ml of the sample to a 500-ml beaker and dilute to about 300 ml with water. Add 2 or 3 drops of methyl orange indicator and titrate with standard sodium hydroxide solution (see A-2.1.4).

A-2.2.2.2 Lead and zinc fluoborates—Transfer 10.0 ml of the sample to a 250-ml beaker and titrate with standard sodium hydroxide solution (see A-2.1.4) to a faint permanent turbidity.

A-2.2.3 Calculation

Free fluoboric acid (HBF_4), $\text{g/l} = V \times N \times 8.8$

where

V = volume in ml of standard sodium hydroxide solution (see **A-2.1.4**) consumed in titration, and

N = normality of standard sodium hydroxide solution used.

A-3. DETERMINATION OF FREE BORIC ACID

A-3.0 General—In fluoboric acid, free boric acid is determined using the titrated solution reserved in **A-2.2.1**. In the case of metal fluoborates, 10.0 ml of the sample is passed through the cation exchange resin column before proceeding with the determination.

A-3.1 Reagents

A-3.1.1 Cation Exchange Resin—Sodium form of sulphonated polystyrene is suitable.

A-3.1.2 Dilute Hydrochloric Acid—approximately 1 N. Dilute 50 ml of concentrated hydrochloric acid (11 N) to 500 ml with water.

A-3.1.3 Phenolphthalein Indicator Solution—prepared by dissolving 0.1 g of phenolphthalein in 100 ml of 60 percent (v/v) rectified spirit.

A-3.1.4 Mannitol

A-3.1.5 Standard Sodium Hydroxide Solution—same as in **A-2.1.3**.

A-3.1.6 Standard Sodium Hydroxide Solution—same as in **A-2.1.4**.

A-3.2 Procedure

A-3.2.1 For Fluoboric Acid—To the titrated solution reserved in **A-2.2.1** add 3 to 4 drops of phenolphthalein indicator solution, 5 g of mannitol and titrate with standard sodium hydroxide solution (see **A-3.1.6**). Let the volume of this standard sodium hydroxide solution consumed be B ml.

A-3.2.1.1 Calculation

Free boric acid (H_3BO_3), $\text{g/l} = (B - 0.5 A) \times 61.84 \times N$

where

A = volume of standard sodium hydroxide solution (**A-2.1.3**) consumed in **A-2.2.1**, and

N = normality of standard sodium hydroxide solution (see **A-2.1.4**).

A-3.2.2 For Metal Fluoborates

A-3.2.2.1 Preparation of cation exchange column—Fill a glass tube (600×25 mm and fitted with a stop-cock and glass wool plug) with the cation exchange resin to a level of 450 mm above the glass wool plug. Run 50 ml of hydrochloric acid through the column at a rate of not more than 1 ml per 5 seconds and wash the column with water until the effluent is free from chloride ions.

A-3.2.2.2 Treatment and determination—Transfer 10.0 ml of the sample of metal fluoborate to a 100-ml volumetric flask and dilute to the mark with water. Transfer a 10.0-ml aliquot to the top of the cation exchange column and allow the eluate to run into a 500-ml conical flask at a rate of not more than 1 ml per 5 seconds. Wash the column with water until the eluate is neutral to litmus. Add 50 ml of calcium chloride solution and a few drops of methyl orange indicator solutions and titrate with standard sodium hydroxide solution to the first sign of colour change from pink to yellow and continue as in A-2.2.1 and A-3.2.1. Calculate free boric acid as in A-3.2.1.1.

A-4. DETERMINATION OF LEAD

A-4.0 General—Lead is determined in lead fluoborate by the volumetric method using magnesium potassium salt of EDTA and in zinc fluoborate by the colorimetric method using dithiozone.

A-4.1 Reagents

A-4.1.1 For Lead Fluoborate

A-4.1.1.1 Ascorbic acid—solid.

A-4.1.1.2 Sodium potassium tartrate—solid.

A-4.1.1.3 Ammonia solution—approximately 8 N. Dilute 50 ml of concentrated ammonia, 35 percent (m/m) (18 N), with 50 ml of water.

A-4.1.1.4 EDTA magnesium potassium salt solution—30 g/l.

A-4.1.1.5 Nitric acid—approximately 8 N. Dilute 50 ml of concentrated nitric acid, 70 percent (m/m) (16 N), with 50 ml of water.

A-4.1.1.6 Potassium cyanide solution—200 g/l.

A-4.1.1.7 EDTA standard solution—0.1 M. Standardize against pure lead by the procedure given in A-4.2.1.1.

A-4.1.1.8 Buffer solution—Dissolve 54 g of ammonium chloride in 350 ml of concentrated ammonia solution, 35 percent (m/m) (18 N), and dilute to 1 litre with water.

A-4.1.1.9 Indicator—Grind in a mortar 100 g of sodium chloride and 0.5 g of eriochrome black T.

A-4.1.2 For Zinc Fluoborate**A-4.1.2.1 Chloroform****A-4.1.2.2 Acetic acid**—glacial (17 M).**A-4.1.2.3 Ammonia solution**—concentrated, 25 percent (*m/m*) (13.5 N).

A-4.1.2.4 Dithizone solution—0.06 g/l. Dissolve 30 mg of dithizone in 25 ml of chloroform and extract twice in a large separating funnel with 100 ml portions of approximately 0.4 N ammonia solution. Discard the chloroform layer and wash the combined aqueous layers with about 20 ml of chloroform. Again discard the chloroform layer. Make the aqueous solution just acid by the addition of concentrated hydrochloric acid, 36 percent (*m/m*) (11 N), and shake with 250 ml of chloroform. Separate the chloroform layer, add a further 250 ml of chloroform and shake again. Separate and combine the two chloroform extracts. Wash with water and store in a dark bottle.

A-4.1.2.5 Hydroxyl ammonium chloride solution—100 g/l. Dissolve 10 g of hydroxyl ammonium chloride in water and dilute to 100 ml.

A-4.1.2.6 Nitric acid—approximately 8 N. Dilute 50 ml of concentrated nitric acid, 70 percent (*m/m*) (16 N) to 100 ml with water.

A-4.1.2.7 Potassium cyanide solution (ammoniacal)—Dissolve 5 g of potassium cyanide and 1 g of sodium sulphite in 400 ml of water and add 350 ml of concentrated ammonia solution, 25 percent (*m/m*) (13.5 N), or 250 ml of concentrated ammonia solution, 35 percent (*m/m*) (18 N).

A-4.1.2.8 Sodium tartrate solution—200 g/l. Dissolve 20 g of sodium tartrate in water and dilute to 100 ml.

A-4.1.2.9 Standard lead solution—Dissolve 40.0 mg of lead nitrate in approximately 0.8 N nitric acid, transfer to a 250-ml volumetric flask, and dilute to the mark. Dilute 25 ml of this solution to 250 ml in a volumetric flask. One millilitre of this solution is equivalent to 0.01 mg of lead (as Pb).

A-4.1.2.10 Thymol blue indicator solution—Warm 0.1 g of thymol blue with 4.3 ml of sodium hydroxide solution (2 g/l) and 5 ml of alcohol until dissolved. Dilute with alcohol (20 percent) to 250 ml.

A-4.2 Procedure

A-4.2.1 For Lead Fluoborate—Transfer 20.0 ml of the sample to a 250-ml volumetric flask and dilute to the mark. Transfer a 20.0-ml aliquot to a 400-ml beaker, make just neutral with ammonia solution and just acid with nitric acid (add nitric acid dropwise to clear any turbidity).

Add 2 g of sodium potassium tartrate, 15 ml of the buffer solution and dilute to 250 ml with water. Add 5 ml of potassium cyanide solution, approximately 0.05 g of ascorbic acid, 10 drops of magnesium potassium EDTA solution and sufficient eriochrome black T indicator to give a pale-magenta colour and titrate with the standard EDTA solution to blue colour, in which there is no trace of red.

Lead (Pb), g/l = ml of 0.1 M EDTA $\times F \times 12.99$

where F is the factor determined by standardization of the EDTA solution as in A-4.2.1.1.

A-4.2.1.1 Standardization of EDTA solution—Weigh accurately about 0.8 g of pure lead metal and dissolve in 10 ml of nitric acid. Neutralize with ammonia solution and add sufficient of nitric acid to just dissolve the lead hydroxide. Add 2 g of sodium potassium tartrate and continue as in A-4.2.1.

A-4.2.2 For Zinc Fluoborate—Dilute 5.0 ml of the sample plus 25 ml of nitric acid to 250 ml in a volumetric flask. Transfer 5.0-ml aliquot to a 100-ml beaker, add 5 ml of water and 1 ml of hydroxylammonium chloride solution and heat to incipient boiling. Cool, add 2 ml of sodium tartrate solution and 1 drop of thymol blue indicator and neutralize with ammonia solution to the blue colour. Transfer to a 200-ml separating funnel with 65 ml of ammoniacal potassium cyanide solution. Add 20.0 ml of dithizone solution and shake for 1 minute. Insert a roll of filter paper in the stem of the funnel and run off the chloroform layer into a 100-ml Nessler cylinder.

A-4.2.2.1 Carry out simultaneously a control test following the same procedure as described in A-4.2.2, using the same quantity of reagents and 5.0 ml of standard lead solution in place of the sample aliquot. The material shall be taken as having satisfied the requirement of the test if the intensity of colour produced with the sample is not deeper than in the control test.

A-5. DETERMINATION OF TIN IN TIN FLUOBORATE

A-5.1 Reagents

A-5.1.1 Hydrochloric Acid—1:2 (v/v).

A-5.1.2 Starch Indicator Solution—Dissolve 0.5 g of soluble starch in 100 ml of boiling water, cool and filter. This solution shall be freshly prepared.

A-5.1.3 Standard Potassium Iodate Solution—Dissolve 6.02 g of potassium iodate, previously dried at 105 to 110°C, in 400 ml of water containing 1 g of sodium hydroxide. Add 30 g of potassium iodide and dilute to 1 litre in a volumetric flask. One millilitre of this solution is equivalent to 0.01 g of tin (as Sn).

A-5.2 Procedure—Transfer 20.0 ml of the sample to a 250-ml volumetric flask and dilute to the mark with water. Transfer 20.0-ml aliquot to a 250-ml beaker, add 10 ml of hydrochloric acid, 2 to 3 ml of starch solution and titrate with standard potassium iodate solution to a permanent blue colour.

A-5.3 Calculation

Tin, g/l = volume of iodate solution (ml) \times 6.25

A-6. DETERMINATION OF COPPER

A-6.0 General—Three methods are prescribed. The electrolytic and volumetric methods are for copper fluoborate and the colorimetric method for tin and zinc fluoborates.

A-6.1 Reagents

A-6.1.1 For Electrolytic Method

A-6.1.1.1 Acetone—See IS: 170-1966*.

A-6.1.1.2 Sodium sulphide solution—100 g/l. Prepare freshly as required by dissolving 5 g of sodium sulphide ($\text{Na}_2\text{S} \cdot 9\text{H}_2\text{O}$) in 50 ml of water.

A-6.1.2 For Volumetric Method

A-6.1.2.1 Ammonium thiocyanate—solid.

A-6.1.2.2 Dilute acetic acid—approximately 2 M. Mix 10 ml of glacial acetic acid (17 M) with 90 ml of water.

A-6.1.2.3 Orthophosphoric acid—90 percent (m/m).

A-6.1.2.4 Potassium iodide

A-6.1.2.5 Sodium carbonate—100 g/l.

A-6.1.2.6 Sodium thiosulphate solution—0.1 N approximately.

A-6.1.2.7 Starch indicator solution—5 g/l. Dissolve 0.5 g of soluble starch in 100 ml of boiling water, cool and filter. This solution shall be freshly prepared.

A-6.1.3 For Colorimetric Method

A-6.1.3.1 Triammonium citrate—solid.

A-6.1.3.2 Acetaldehyde solution—400 g/l, reagent solution 40 percent by volume in methanol. As the boiling point of acetaldehyde is 21°C, mix carefully in the cold.

*Specification for acetone (first revision).

A-6.1.3.3 *Ammonia solution* — concentrated, 35 percent (*m/m*) (18 N).

A-6.1.3.4 *Citric acid monohydrate solution* — 500 g/l.

A-6.1.3.5 *Oxalyldihydrazide solution* — 2.5 g/l. Heat slightly to dissolve.

A-6.1.3.6 *Standard copper solution* — Dissolve 0.3928 g of copper sulphate ($\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$) in sufficient water. Transfer quantitatively to a 1-litre volumetric flask. Make up the volume with water and mix. Transfer 25.0-ml aliquot of this solution to a 250-ml volumetric flask, dilute to the mark with water and mix. One millilitre of this solution is equivalent to 0.01 mg of copper (as Cu).

A-6.2 Procedure

A-6.2.1 *Electrolytic Method* — Dilute 25.0 ml of the sample to 250 ml with water in a volumetric flask. Transfer a 50.0-ml aliquot to a 400-ml beaker and dilute to about 170 ml with water. Insert the tared cathode and anode and cover with a split watch glass. Electrolyze using a current of 2.5 to 3 A with a rotating cathode or a magnetic stirrer until the solution is colourless. Rinse the watch glass and the inner walls of the beaker with water and continue the electrolysis for a further 10 minutes.

A-6.2.1.1 When the electrolysis is complete, rinse the electrodes and without interrupting the current wash the cathode with a fine stream of water. Reserve the filtrate for determination of silica (see A-8.2.4). Switch off the current, remove the cathode, rinse it once with water and twice with acetone. Dry at 105°C and weigh.

NOTE — Confirm the complete deposition of copper by mixing one drop of electrolyte with one drop of sodium sulphide solution on a white tile. A black precipitate of copper sulphide indicates incomplete electrolysis.

A-6.2.1.2 Calculation

Copper, g/l = Increase in mass of cathode (g) \times 200

A-6.2.2 *Volumetric Method* — Transfer a 10.0-ml aliquot (see A-6.2.1) to a 250-ml beaker. Add 2 or 3 drops of orthophosphoric acid and neutralize to a slight permanent turbidity with the sodium carbonate solution. Add dilute acetic acid until the precipitate just dissolves and add about 1 g of potassium iodide. Titrate with the standard sodium thiosulphate solution adding 2 or 3 ml of starch indicator when the solution becomes pale-yellow in colour. Towards the end of the titration, add about 1.5 g of ammonium thiocyanate and complete the titration.

A-6.2.2.1 Calculation

Copper, g/l = $V \times N \times 63.57$

where

V = volume in ml of standard sodium thiosulphate solution used, and

N = normality of standard sodium thiosulphate solution used.

A-6.2.3 Colorimetric Method

A-6.2.3.1 For stannous fluoborate — Transfer 10.0 ml of the sample to a 100-ml volumetric flask and dilute to the mark with water. Transfer 10.0-ml aliquot to a 50-ml Nessler cylinder, add 10 g of triammonium citrate and 4 ml of ammonium hydroxide. Cool and add 20 ml of a mixture of 1 volume of acetaldehyde solution and 1 volume of oxalyldihydrazide solution that has been allowed to stand for 2 hours and filtered, if necessary. Swirl gently, dilute to the mark and mix. Carry out simultaneously a control test following the same procedure as above using the same quantity of reagents and 5.0 ml of the standard copper solution in place of the sample aliquot. The material shall be taken as having satisfied the requirement of the test if the intensity of colour produced with the sample is not deeper than that in the control test.

A-6.2.3.2 For zinc fluoborate — Transfer 5.0 ml of the sample to a 100-ml volumetric flask and dilute to the mark with water. Transfer 5.0 ml aliquot to a 50-ml Nessler cylinder, and add 2 ml of citric acid solution, 5.5 ml of ammonium hydroxide and continue as described in **A-6.2.3.1**.

A-7. DETERMINATION OF SULPHATE**A-7.1 Reagents**

A-7.1.1 Barium Chloride Solution — 100 g/l.

A-7.1.2 Concentrated Hydrochloric Acid — See IS : 265-1962*.

A-7.1.3 Dilute Hydrochloric Acid — approximately 3 N. Dilute 30 ml of concentrated hydrochloric acid to 100-ml with water.

A-7.2 Procedure

A-7.2.1 For Fluoboric Acid and Metal Fluoborates other than of Sodium — Transfer 10.0 ml of the sample to a platinum dish. Evaporate on a water-bath until fumes are no longer evolved. Add 10 ml of water and evaporate again until fumes are no longer evolved. Dissolve the residue in the dish with 20 ml of dilute hydrochloric acid, transfer to a 500-ml beaker and dilute to about 300 ml with water. Heat to boiling and add 10 ml of barium chloride solution slowly over a period of 2 to 4 minutes in a very fine stream. Allow to stand on the edge of a hot-plate for 1 hour and then overnight at room temperature. Filter on a previously weighed sintered glass crucible (G No. 4) and wash well with water. Dry to constant mass at 110°C. Cool and weigh the precipitate as barium sulphate.

A-7.2.1.1 Calculation

Sulphate (SO_4), g/l = Mass of barium sulphate (g) \times 41.15

*Specification for hydrochloric acid (revised).

A-7.2.2 For Sodium Fluoborate—Weigh accurately about 10 g of the sample, dissolve in 250-ml of water in a 500-ml beaker. Add 5 ml of concentrated hydrochloric acid, heat to boiling and add 25 ml of barium chloride solution slowly over a period of 2 to 4 minutes in a very fine stream and continue as in **A-7.2.1**.

A-7.2.2.1 Calculation

$$\text{Sulphate (SO}_4\text{), percent by mass} = \frac{M_1}{M} \times 41.15$$

where

M = mass in g of the sample taken, and

M_1 = mass in g of the barium sulphate precipitate obtained.

A-8. DETERMINATION OF SILICA

A-8.0 General—Silica is determined colorimetrically as silicon molybdate.

A-8.1 Reagents

A-8.1.1 Ammonium Molybdate—100 g/l. Dissolve 10 g of ammonium molybdate in 100 ml of water.

A-8.1.2 Bromine Water—saturated solution of bromine in water.

A-8.1.3 Sodium Nitrite Solution—100 g/l. Dissolve 10 g of sodium nitrite in 100 ml of water.

A-8.1.4 Dilute Sulphuric Acid—approximately 1 N. Dilute 28 ml of concentrated sulphuric acid, 98 percent (m/m) (36 N), to 1 litre with water.

A-8.1.5 Standard Silica Solution—Fuse 0.1 g pure anhydrous silica (SiO_2) with 2 g of anhydrous sodium carbonate in a platinum crucible, dissolve in water and dilute to 1 litre. One millilitre of this solution is equivalent to 0.1 mg of silica (as SiO_2).

A-8.2 Procedure

A-8.2.1 For Fluoboric Acid and Zinc Fluoborate—Transfer 5.0 ml of the sample to a 250-ml volumetric flask and dilute to the mark with water. Transfer 5.0 ml aliquot of this solution to 100-ml Nessler cylinder, add 70 ml of water, 10 ml of dilute sulphuric acid, 5 ml of ammonium molybdate and dilute to the mark with water. Allow to stand for 15 minutes. Carry out simultaneously a control test following the same procedure, using the same quantity of reagents as with the sample and 10 ml of standard silica solution in place of the sample. The colour produced with the sample shall not be deeper than that produced in the control test.

A-8.2.2 For Lead Fluoborate — Transfer 5.0 ml of the sample to a 250-ml volumetric flask. Add 50 ml of water, 20 ml of dilute sulphuric acid and dilute to the mark with water. Allow to settle, filter through a dry filter paper, discarding the first 10 ml of the solution. Transfer 5.0 ml of the clear filtrate to a 100-ml Nessler cylinder, add 10 ml of dilute sulphuric acid and continue as in **A-8.2.1**.

A-8.2.3 For Stannous Fluoborate — Transfer 5.0 ml of the sample to a 250-ml volumetric flask and dilute to the mark with water. Transfer 5.0-ml aliquot of this solution to a 100-ml Nessler cylinder, add 10 ml of dilute sulphuric acid, oxidize with bromine water until there is a slight excess. Just destroy the excess of bromine by addition of a few drops of sodium nitrite solution and continue as in **A-8.2.1**.

A-8.2.4 For Copper Fluoborate — Transfer the solution reserved in **A-6.2.1.1** to a 500-ml volumetric flask and dilute to the mark with water. Transfer 10.0-ml aliquot of this solution to a 100-ml Nessler cylinder, add 10 ml of dilute sulphuric acid and continue as in **A-8.2.1**.

A-8.2.5 For Sodium Fluoborate — Weigh accurately about 5.00 g of the sample and dissolve in 100 ml of water. Transfer to a 250-ml volumetric flask and dilute to the mark with water. Transfer 5.0-ml aliquot of this solution to a 100-ml Nessler cylinder and continue as in **A-8.2.1**, using 8.0 ml of the standard silica solution in the control test.

A-9. DETERMINATION OF IRON

A-9.1 Reagents

A-9.1.1 Aluminium Solution — Dissolve 2.34 g of aluminium sulphate $[\text{Al}_2(\text{SO}_4)_3 \cdot 16\text{H}_2\text{O}]$ in 100 ml of water. One millilitre of this solution is equivalent to two milligrams of aluminium (as Al).

A-9.1.2 Hydroxylammonium Chloride Solution — 100 g/l. Dissolve 10 g of hydroxylammonium chloride in 100 ml of water.

A-9.1.3 o-Phenanthroline Solution — 2.5 g/l. Dissolve 0.25 g of o-phenanthroline in 100 ml of hot water and allow to cool.

A-9.1.4 Buffer Solution — Dissolve 270 g of sodium acetate (anhydrous) in 500 ml of water, add 240 ml of acetic acid, glacial (17 M), and dilute to 1 litre.

A-9.1.5 Dilute Nitric Acid — approximately 8 N. Dilute 50 ml of concentrated nitric acid, 70 percent (m/m) (16 N), to 100 ml with water.

A-9.1.6 Ammonium Hydroxide Solution — approximately 9 N. Dilute 50 ml of concentrated ammonium hydroxide, 35 percent (m/m) (18 N), to 100 ml with water.

A-9.1.7 Glacial Acetic Acid**A-9.1.8 Ammonium Nitrate Solution** — 10 g/l.

A-9.1.9 Standard Iron Solution — Dissolve 7.02 g of ferrous ammonium sulphate $[(\text{NH}_4)_2\text{SO}_4 \cdot \text{FeSO}_4 \cdot 6\text{H}_2\text{O}]$ in water containing 10 ml of concentrated sulphuric acid and dilute to 1000 ml with water in a 1000-ml volumetric flask. Dilute 5.0-ml aliquot of this solution to 100 ml in a 100-ml volumetric flask. One millilitre of this diluted solution is equivalent to 0.05 mg of iron (as Fe).

A-9.2 Procedure

A-9.2.1 For Fluoboric Acid and Lead and Tin Fluoborates — Transfer 5.0 ml of the sample to a 100-ml volumetric flask in the case of fluoboric acid and lead fluoborate and to a 250-ml volumetric flask in the case of tin fluoborate. Dilute to the mark with water. Transfer 5.0-ml aliquot of this solution to a 100-ml beaker, neutralize to litmus with ammonium hydroxide and make just acidic with glacial acetic acid. Add 15 ml of the buffer solution and 2 ml of hydroxylammonium chloride solution and heat to incipient boiling. Cool, add 10 ml of *o*-phenanthroline solution, transfer to a 50-ml Nessler cylinder and dilute to the mark with water. Carry out simultaneously, a control test following the same procedure, using the same quantity of reagents as with the sample and the following amounts of standard iron solution in place of the sample:

| <i>Name of Material</i> | <i>Volume of Standard Iron Solution</i> ml |
|-------------------------|---|
| Fluoboric acid | 1 |
| Lead fluoborate | 2 |
| Tin fluoborate | 4 |

The colour produced with the sample shall not be deeper than that produced in the control test.

A-9.2.2 For Zinc Fluoborate and Copper Fluoborate — Transfer 5.0 ml of the sample to a 100-ml volumetric flask in the case of zinc fluoborate and 250-ml volumetric flask in the case of copper fluoborate. Dilute to the mark with water. Transfer 5.0-ml aliquot to a 100-ml beaker, add 80 ml of water and 2 ml of aluminium solution, neutralize with ammonium hydroxide solution, and add sufficient excess to redissolve the zinc and copper hydroxide. Heat to coagulate, filter through hardened filter paper (Whatman No. 541) and wash with hot ammonium nitrate solution. Wash the precipitate off the filter paper into the original beaker and dissolve in 5 ml of dilute nitric acid and evaporate to a volume of less than 10 ml. Cool, neutralize to litmus with ammonium hydroxide, make just acidic with glacial acetic acid and continue as in **A-9.2.1** using 5.0 and 4.0 ml of standard iron solution for zinc and copper fluoborates respectively in the control test.

A-9.2.3 For Sodium Fluoborate—Weigh accurately about 1.00 g of the sample and dissolve in 50 ml of water. Transfer to a 100-ml volumetric flask and dilute to the mark with water. Transfer 5.0-ml aliquot of this solution to a 100-ml beaker, neutralize to litmus with ammonium hydroxide, make just acidic with glacial acetic acid and continue as in **A-9.2.1**, using 5.0 ml of the standard iron solution in the control test.

A-10. DETERMINATION OF HEAVY METALS OTHER THAN IRON

A-10.1 Reagents

A-10.1.1 Hydrogen Sulphide Solution—A fresh solution prepared by passing a current of washed hydrogen sulphide gas through water to saturation.

A-10.1.2 Standard Lead Solution—Freshly prepared by dissolving 0.16 g of lead nitrate in 5 ml of concentrated nitric acid (70 percent m/m) (16 N) and 100 ml of water and finally diluting to 1 000 ml. One millilitre of this solution is equivalent to 0.1 mg of lead (as Pb).

A-10.2 Procedure—Transfer 5.0 ml of the sample to a 250-ml volumetric flask and dilute to the mark with water. Transfer 5.0-ml aliquot to a 50-ml Nessler cylinder. Add 5 ml of hydrogen sulphide solution, dilute to 50 ml. The colour produced shall be not deeper than that produced by 1.0 ml of the standard lead solution under the same conditions in the control test.

A-11. DETERMINATION OF CHLORIDE

A-11.1 Reagents

A-11.1.1 Ferric Nitrate Solution—150 g/l. Dissolve 15 g of ferric nitrate in 90 ml of water and add 10 ml of concentrated nitric acid, 70 percent (m/m) (16 N).

A-11.1.2 Nitric Acid—approximately 8 N. Dilute 50 ml of concentrated nitric acid, 70 percent (m/m) (16 N), to 100 ml with water.

A-11.1.3 Standard Silver Nitrate Solution—0.05 N approximately.

A-11.1.4 Standard Silver Nitrate Solution—0.02 N approximately.

A-11.1.5 Standard Ammonium Thiocyanate Solution—0.05 N approximately.

A-11.1.6—Standard Ammonium Thiocyanate Solution—0.02 N approximately.

NOTE—It is important that normalities of silver nitrate solutions match those of ammonium thiocyanate solutions for correct estimation.

A-11.2 Procedure

A-11.2.1 For Fluoboric Acid—Transfer 10.0 ml of the solution to a 150-ml beaker, add 50 ml of water, 15 ml of nitric acid and 4 ml of the ferric nitrate solution. Add 10.0 ml of silver nitrate solution (0.02 N) and titrate with ammonium thiocyanate solution (0.02 N) to the first colour change from white to orange.

A-11.2.2 For Tin Fluoborate—Transfer 10.0 ml of the sample to a 250-ml beaker, add 90 ml of water, 10 ml of nitric acid, boil for 5 minutes, cool to room temperature and add 4 ml of ferric nitrate solution. Add 10.0 ml of silver nitrate solution (0.05 N) and titrate immediately with ammonium thiocyanate solution (0.05 N) to the first colour change from white to orange.

A-11.2.3 For Sodium Fluoborate—Weigh accurately about 3 g of the sample and dissolve in 100 ml of water in 250-ml beaker. Add 10 ml of nitric acid and continue as in A-11.2.2.

A-11.3 Calculation

A-11.3.1 For Fluoboric Acid and Tin Fluoborate

$$\text{Chloride (as Cl), g/l} = (10 - V) \times N \times 3.545$$

where

V = volume in ml of ammonium thiocyanate solution consumed, and

N = normality of silver nitrate solution used.

A-11.3.2 For Sodium Fluoborate

$$\text{Chloride (as Cl), percent by mass} = \frac{(10 - V) N \times 3.545}{M}$$

where

V = volume in ml of ammonium thiocyanate solution consumed,

N = normality of silver nitrate solution used, and

M = mass in g of the sample taken for the test.

A-12. DETERMINATION OF ZINC

A-12.1 Reagents

A-12.1.1 Buffer Solution—Add 54 g of ammonium chloride to 350 ml of ammonia solution, 35 percent (m/m) (18 N), and dilute to 1 litre with water.

A-12.1.2 Indicator Solution—0.4 g eriochrome black T and 3 g of hydroxylammonium chloride dissolved in 100 ml of ethanol (95 percent by volume).

A-12.1.3 Concentrated Hydrochloric Acid—See IS: 265-1962*.

A-12.1.4 Triethanolamine Solution—10 percent (*v/v*).

A-12.1.5 Potassium Cyanide Solution—200 g/l.

A-12.1.6 Formaldehyde Solution—10 percent (*v/v*). Dilute 1 volume of formaldehyde, 400 g/l, with 9 volumes of water.

A-12.1.7 Standard EDTA (Disodium Salt Solution)—0.1 M approximately. Standardize as follows:

Weigh accurately about 2 g of pure zinc metal and dissolve in 20 ml of water and 5 ml of concentrated hydrochloric acid. Transfer to a 250-ml volumetric flask and dilute to the mark with water. Transfer 25.0-ml aliquot to a 500-ml beaker and continue as in **A-12.2**.

Then, factor of standardization (*F*) is given by the following:

$$F = \frac{\text{Mass of zinc metal (g)}}{10 \times \text{volume of 0.1 M EDTA used (ml)} \times 0.006538}$$

A-12.2 Procedure—Transfer 25.0 ml of the sample to a 500-ml volumetric flask and dilute to the mark with water. Transfer 25.0 ml of this solution to a 400-ml beaker and neutralize to litmus with triethanolamine solution. Add 10 ml of the buffer solution and dilute to 200 ml. Add 10 ml of potassium cyanide solution, 1 ml of eriochrome black T indicator solution, 20 ml of formaldehyde solution and titrate with the standard EDTA solution to the appearance of blue colour. Add a further 10 ml of formaldehyde solution and continue the titration to the removal of the last traces of red colour in the solution. Confirm that the end point is permanent by the addition of a further 5 ml of formaldehyde solution.

A-12.3 Calculation

$$\text{Zinc, g/l} = \text{volume of 0.1 M EDTA used (ml)} \times F \times 5.23$$

A-13. DETERMINATION OF SODIUM FLUOBORATE

A-13.0 General—Sodium fluoborate is estimated gravimetrically by precipitation as lead chlorofluoride.

A-13.1 Reagents

A-13.1.1 Bromophenol Blue Indicator Solution—Dissolve 0.1 g in 100 ml of rectified spirit.

*Specification for hydrochloric acid (*revised*).

A-13.1.2 Sodium Chloride Solution — 10 percent (*m/v*).

A-13.1.3 Dilute Nitric Acid — approximately 8 N. Dilute 50 ml of concentrated nitric acid, 70 percent (*m/m*) (16 N), to 100 ml with water.

A-13.1.4 Sodium Hydroxide Solution — approximately 1 N.

A-13.1.5 Concentrated Hydrochloric Acid — See IS: 265-1962*.

A-13.1.6 Lead Nitrate

A-13.1.7 Sodium Acetate

A-13.1.8 Lead Chlorofluoride Wash Solution — saturated.

A-13.2 Procedure — Weigh accurately about 1 g of the sample. Transfer to a 400-ml beaker and dissolve in 100 ml of water. Add 2 drops of bromophenol blue indicator solution, 3 ml of sodium chloride solution and dilute to about 250 ml with water. Add dilute nitric acid till the colour just changes to yellow. Then add sodium hydroxide solution slowly till the colour changes back to blue. Add 1 ml of concentrated hydrochloric acid, 5 g of lead nitrate. Place on a water-bath and stir well to dissolve the salts. Add 5 g of sodium acetate with vigorous stirring, digest for 30 minutes over a water-bath with occasional stirring and allow to stand overnight.

A-13.2.1 Filter the solution with precipitate through a sintered glass crucible (G No. 4), wash once with cold water and then four times with lead chlorofluoride wash solution. Wash once more with cold water and dry the crucible with residue at 130 to 140°C to a constant mass.

A-13.3 Calculation

$$\text{Sodium fluoborate (as NaBF}_4 \text{), percent by mass} = \frac{M_1}{M} \times 10.49$$

where

M_1 = mass in g of the residue, and

M = mass in g of the sample taken for the test.

A-14. DETERMINATION OF VOLATILE MATTER

A-14.1 Procedure — Weigh accurately about 1 g of the sample in a tared crucible and heat at $105 \pm 2^\circ\text{C}$ to a constant mass and calculate the percentage of loss in mass.

*Specification for hydrochloric acid (revised).

APPENDIX B

(Clause 4.2)

SAMPLING OF SODIUM FLUOBORATE FOR ELECTROPLATING

B-1. SCALE OF SAMPLING

B-1.1 Lot — All the containers in a single consignment of the material drawn from a single batch of manufacture shall constitute a lot.

B-1.2 For ascertaining the conformity of material in the lot to the requirements of this specification, samples shall be tested from each lot separately. The number of containers to be selected at random from the lot shall depend on the size of the lot and shall be in accordance with Table 2.

TABLE 2 SCALE OF SAMPLING

| LOT SIZE (NUMBER OF CONTAINERS) | SAMPLE SIZE |
|---------------------------------------|--------------|
| (<i>N</i>) | (<i>n</i>) |
| Up to 15 | 2 |
| 16 „ 25 | 3 |
| 26 „ 50 | 4 |
| 51 „ 100 | 5 |
| 101 „ 150 | 6 |
| 151 and above | 8 |

B-1.3 In order to ensure the randomness of selection, random sampling procedure given in IS : 4905-1968* may be followed.

B-2. TEST SAMPLE AND REFEREE SAMPLE

B-2.1 From each of the containers selected according to **B-1.2**, a representative portion of the material shall be drawn from different parts of the container. The total quantity so drawn from each of the containers shall be approximately equal to thrice the quantity required for testing purposes.

B-2.2 From each of the individual samples a small but equal quantity of material shall be taken. Such portions shall be thoroughly mixed to give a composite sample.

*Methods of random sampling.

B-2.3 The material constituting each of the individual samples as well as composite sample shall be stored separately with full identification particulars.

B-2.4 The referee samples consisting of composite sample and a set of individual samples shall bear the seals of both the purchaser and the supplier and shall be kept at a place agreed to between the two. This shall be used in case of any dispute between the two.

B-3. NUMBER OF TESTS

B-3.1 Test for sodium fluoborate content shall be conducted on individual samples.

B-3.2 The remaining characteristics shall be tested on the composite sample.

B-4. CRITERIA FOR CONFORMITY

B-4.1 For Individual Samples — For sodium fluoborate content, the test results shall be noted and their mean (\bar{x}) and the range (R) being the difference between the maximum and minimum of test results, shall be computed. For declaring the conformity of the lot in respect of sodium fluoborate content, ($\bar{x} - 0.4 R$) shall be greater than or equal to 96.6.

B-4.2 For declaring the conformity of the lot to the requirements of all other characteristics, the test results of the composite sample shall satisfy the relevant requirements given in Table 1.

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